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ATTORNEY'S DOCKET NUMBER JC10 Rec'd PCT/PTO 17 DEC 2001

2541-000011

U.S. APPLICATION NO. (If known, see 37 CFR 1.5

10/018757

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/FR00/01828	29 June 2000 (29.06.00)	30 June 1999 (30.06.99)

TITLE OF INVENTION
PROCESS FOR MAKING A THIN FILM BY APPLYING PRESSURE

APPLICANT(S) FOR DO/EO/US ASPAR, Bernard; BRUEL, Michel and MORICEAU, Hubert

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. [X] This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. [ ] This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. [ ] This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4. [X] The US has been elected by the expiration of 19 months from the priority date (Article 31).
5. [X] A copy of the International Application as filed (35 U.S.C. 371(c)(2))
   - a. [ ] is attached hereto (required only if not communicated by the International Bureau).
   - b. [X] has been communicated by the International Bureau.
   - c. [ ] is not required, as the application was filed in the United States Receiving Office (RO/US).
6. [X] An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
   - a. [X] is attached hereto. 20 pages plus 1 page Abstract
   - b. [ ] has been previously submitted under 35 U.S.C. 154(d)(4).
7. [ ] Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
   - a. [ ] are attached hereto (required only if not communicated by the International Bureau).
   - b. [ ] have been communicated by the International Bureau.
   - c. [ ] have not been made; however, the time limit for making such amendments has NOT expired.
   - d. [ ] have not been made and will not be made.
8. [ ] An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371 (c)(3)).
9. [ ] An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. [X] An English language translation of the annexes of the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)). English translation of amended sheets of IPER -3 pages

Items 11 to 20 below concern document(s) or information included:

11. [ ] An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. [ ] An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. [X] A FIRST preliminary amendment. 5 pages
14. [ ] A SECOND or SUBSEQUENT preliminary amendment.
15. [ ] A substitute specification.
16. [ ] A change of power of attorney and/or address letter.
17. [ ] A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 U.S.C. 1.821 - 1.825.
18. [ ] A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. [ ] A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. [X] Other items or information:

* Priority claimed from French Patent Application No. 08379 filed 30 June 1999.
Application Data Sheet (2 pgs.), 1 sheet of Formal Drawings (Figs 1 - 4), copy of International Search Report (in French with English translation), copy of International Preliminary Examination Report (in French), and return postcard.

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U S APPLICATION NO (if known, see 37 CFR 1.5) 10/018757	INTERNATIONAL APPLICATION NO. PCT/FR00/01828	ATTORNEY'S DOCKET NUMBER 2541-000011

21. [X] The following fees are submitted:

BASIC NATIONAL FEE (37 CFR 1.492 (a) (1) - (5)):

	CALCULATIONS	PTO USE ONLY
Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO **$1040.00**		
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO **$890.00**		
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO **$740.00**		
International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) **$710.00**		
International preliminary examination fee (37 CFR 1.482) paid to USPTO and all claims satisfied provisions of PCT Article 33(1)-(4) **$100.00**		
ENTER APPROPRIATE BASIC FEE AMOUNT =	$ 890.00	
Surcharge of **$130.00** for furnishing the oath or declaration later than [ ] 20 [ ] 30 months from the earliest claimed priority date (37 CFR 1.492(e)).	$ 0.00	

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE	$
Total claims	20 - 20 =	0	x **$18.00**	$ 0.00
Independent claims	1 - 3 =	0	x **$84.00**	$ 0.00
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			+ **$280.00**	$ 0.00

	CALCULATIONS	PTO USE ONLY
TOTAL OF ABOVE CALCULATIONS =	$ 890.00	
[ ] Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2. +	$ 0.00	
SUBTOTAL =	$ 890.00	
Processing fee of **$130.00** for furnishing the English translation later than [ ] 20 [ ] 30 months from the earliest claimed priority date (37 CFR 1.492(f)).	$ 0.00	
TOTAL NATIONAL FEE =	$ 890.00	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). **$40.00** per property +	$ 0.00	
TOTAL FEES ENCLOSED =	$ 890.00	
	Amount to be refunded:	$
	charged:	$

a. [X] A check in the amount of $ 890.00 to cover the above fees is enclosed.

b. [ ] Please charge my Deposit Account No. ______ in the amount of $ ______ to cover the above fees. A duplicate copy of this sheet is enclosed.

c. [X] The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 08-0750. A duplicate copy of this sheet is enclosed.

d. [ ] Fees are to be charged to a credit card. **WARNING:** Information on this form may become public. **Credit card information should not be included on this form.** Provide credit card information and authorization on PTO-2038.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137 (a) or (b)) must be filed and granted to restore the application to pending status.

SEND ALL CORRESPONDENCE TO:

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Linda M. Deschere
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34,811
REGISTRATION NUMBER

Dated: December 17, 2001

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PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application No.: Not yet assigned – 371 of PCT/FR00/01828

Filing Date: Herewith

Applicant: ASPAR, et al.

Title: PROCESS FOR MAKING A THIN FILM BY APPLYING PRESSURE

Attorney Docket: 2541-00011

Box PCT
Hon. Commissioner of Patents and Trademarks
Washington, D.C. 20231

FIRST PRELIMINARY AMENDMENT

Sir:

Applicants herewith submit this Preliminary Amendment to the application filed herewith, for consideration prior to the calculation of the filing fee, as follows:

IN THE CLAIMS

Please cancel claims 1 - 20 without prejudice and replace with new claims 21 – 40.

21. (NEW) A process for making a thin film starting from a substrate of a solid material having a plane face comprising:

the implantation of gaseous compounds in the substrate to make a layer of micro-cavities at a depth from the plane face corresponding to the thickness of the required thin film, the gaseous compounds being implanted under conditions that could weaken the substrate at the layer of micro-cavities; and

partial or total separation of the thin film from the rest of the substrate, this separation comprising a step in which thermal energy is added and pressure is applied to the plane face.

22. (NEW) The process according to claim 21, in which the pressure is a gas pressure.

23. (NEW) The process according to claim 21, in which the pressure is a mechanical pressure.

24. (NEW) The process according to claim 23, in which the mechanical pressure is generated using a piston.

25. (NEW) The process according to claim 21, in which the pressure is applied locally on the plane face.

26. (NEW) The process according to claim 21, in which the pressure is applied uniformly on the plane face.

27. (NEW) The process according to claim 21, also comprising bonding of a thickener onto the plane face, after implantation of the gaseous compounds.

28. (NEW) The process according to claim 27, in which the thickener is composed of a wafer.

29. (NEW) The process according to claim 28, in which the wafer is bonded by molecular bonding with the plane face.

30. (NEW) The process according to claim 27, in which the thickener is formed by deposition of one or several materials.

31. (NEW) The process according to claim 27, in which the pressure is applied through the thickener.

32. (NEW) The process according to claim 21, in which the pressure is adjusted during the coalescence of at least part of the micro-cavities, to remain slightly above a pressure called the limiting pressure, below which blisters appear on the plane face and above which blisters do not appear on the plane face.

33. (NEW) The process according to claim 21, in which coalescence is performed such that the thin film is separated from the rest of the substrate by pulling them apart.

34. (NEW) The process according to claim 21, in which the thin film is separated from the rest of the substrate by application of a heat treatment and, optionally, by mechanical forces.

35. (NEW) The process according to claim 21, in which the substrate used as the initial substrate is a substrate that has already been used to produce a thin film according to the process.

36. (NEW) The process according to claim 35, in which the previously used substrate is polished to provide a new plane face.

37. (NEW) The process according to claim 21, in which the substrate supports one or several homogeneous and/or heterogeneous layers on the side having the plane face.

38. (NEW) The process according to claim 21, in which the substrate comprises semi-conducting material, at least on the side having the plane face.

39. (NEW) The process according to claim 21, in which the substrate comprises all or part of at least one electronic device and/or at least one electro-optical device, on the side having the plane face.

40. (NEW) The process according to claim 21, in which the separation of the thin film is delayed by the application of an additional step that consists of applying an additional pressure onto the thin film.

REMARKS

After entry of this amendment, claims 21 – 40 are pending in the application. Claims 1 – 20 have been cancelled without prejudice. Claims 21 - 40 have been added in this amendment.

It is submitted that this Amendment has antecedent basis in the application as originally filed, including the specification, claims and drawings, and that this amendment does not add any new subject matter to the application. Consideration of the application as amended is requested.

Respectfully submitted,

Dated: December 15, 2001

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PROCESS FOR MAKING A THIN FILM BY APPLYING PRESSURE

DESCRIPTION

Technical field

This invention relates to a process for making a thin film of solid material. In particular it relates to the production of a thin film made of a semi-conducting material, for example such as silicon.

State of prior art

Document FR-A-2 681 472 (corresponding to American patent 5 374 564) discloses a process for manufacturing thin films made of a semi-conducting material. This document divulges that implantation of a rare gas and/or hydrogen in a substrate made of a semi-conducting material could create a layer that could contain micro-cavities or micro-bubbles (or platelets) at a depth approximately equal to the average penetration depth of the implanted ions. The implanted face of this substrate is brought into intimate contact with a support acting as a stiffener. Furthermore, heat treatment can be applied at a sufficiently high temperature to induce an interaction (or coalescence) between the micro-cavities or micro-bubbles causing a separation of the semi-conducting substrate into two parts, namely a thin semi-conducting film bonding to the stiffener, and secondly the rest of the semi-conducting substrate. The separation takes place at the location at which the micro-cavities or micro-bubbles are present, in other words along the micro-

cavities layer. The heat treatment is such that the interaction between the micro-bubbles or the micro-cavities created by implantation causes a separation between the thin film and the rest of the substrate. Therefore, there is a transfer of a thin film from an initial substrate as far as a stiffener that acts as a support for this thin film.

This process may also be applied to the manufacture of a thin film made of solid material other than a semi-conducting material (a conducting or dielectric material) that may or may not be crystalline. This film may be single layer or multi-layer.

Thus, the implantation of gaseous compounds can create in-depth cavities or micro-bubbles or micro-cracks that will form a weakened layer close to the depth at which the ions stop. The implanted zone is more or less fragile depending on the nature and implantation conditions. They are chosen such that the implanted surface of the substrate is not deformed in any way. If any deformations in this surface occur in the form of blisters, these deformations will cause excessive weakening of the implanted zone.

Document FR-A-2 681 472 describes how, in order to transfer a thin film onto a support, the implanted substrate and the support (or stiffener) have to be bonded together before causing separation of the thin film from its original substrate, this separation possibly being caused by a heat treatment and/or a mechanical treatment (as described in document FR-A-2 748 851). Bonding is achieved by putting the implanted

substrate and the support into intimate contact by means of molecular bonding, or a glue or an intermediate compound (insulating layer, conducting layer, etc.). This bonding is only possible if there are no deformations on the implanted surface, and therefore if no blisters have occurred.

In some cases, it is impossible to bond the implanted substrate and the support acting as a stiffener, particularly due to different coefficients of thermal expansion. It is also possible that the bonding forces are not sufficient to cause the stiffening effect. Therefore a thin film, for example a mono-crystalline film, can be obtained on any support using a process derived from the process divulged by document FR-A-2 681 472, for example the process divulged by document FR-A-2 738 671 (corresponding to American patent 5 714 395). According to this process, the implanted gaseous compounds must be at a sufficient depth and/or a layer of a material able to make the structure sufficiently rigid to obtain separation at the implanted zone must be deposited after the implantation step, in order to separate the thin film from its original substrate. The film obtained is then self supporting.

For the two processes mentioned above, the surface roughness of the thin film after transfer is variable depending on the implantation and/or separation conditions (heat and/or mechanical treatment) used to obtain this separation. In this case it may be useful to further weaken the zone containing the cavities. Separation would then be easier than in the normal

case, in other words separation would be possible by applying lower mechanical forces and/or a smaller thermal budget. This is particularly useful for structures composed of materials with different coefficients of thermal expansion and for which there are limiting heating temperatures.

The various means of weakening the implanted zone include an increase in the dose of implanted gaseous compounds and/or carrying out a heat treatment that may correspond to the heat treatment divulged in document FR-A-2 681 472. However, as mentioned above, the implanted dose and/or the thermal budget need to be limited before the bonding step in order to prevent deformations of the implanted surface.

Thus, there is no acceptable means of further weakening the implanted zone before applying the separation step. The existence of such a means would make it possible to reduce thermal budgets and/or the mechanical forces necessary for separation. Thus, thin films could be transferred onto supports that cannot resist high temperatures, by using the process divulged in document FR-A-2 681 472. It would also be possible to more easily separate thick films using the process described in document FR-A-2 738 671. These thick films could then be transferred onto any type of support, even supports for which it would be impossible to obtain high bonding forces between the film and the support. Furthermore, increased weakening of the implanted zone would make it possible to reduce the roughness of the free surface of the film after transfer, while encouraging fracture.

Therefore, the problem that arises is to further weaken the implanted zone without inducing any blisters on the implanted surface of the original substrate.

Presentation of the invention

The invention provides a solution to this problem. It is proposed to apply pressure on the implanted face of the substrate, at least during part of the coalescence of micro-cavities, in order to facilitate this coalescence and prevent implanted gaseous compounds from escaping from the substrate. The result is that weakening is increased.

Therefore, the purpose of the invention is a process for making a thin film from a substrate of a solid material with a plane face, comprising:

- the implantation of gaseous compounds in the substrate to form a layer of micro-cavities located at a depth from the said plane face corresponding to the thickness of the required thin film, the gaseous compounds being implanted under conditions that can weaken the substrate at the layer of micro-cavities,
- partial or complete separation of the thin film from the rest of the substrate, this separation comprising a step in which thermal energy is input and in which pressure is applied to the said plane face.

The "Mechanistic Studies of Silicon Wafer Bonding and Layer Exfoliation" document by M.K. WELDON et al., published in Electrochemical Society Proceedings, volume 97-36, specifies that application of a compression stress on a glued structure composed of an implanted substrate and a stiffener is a means of

closing micro-cracks and preventing exfoliation, while a uniform external tension can cause exfoliation at a lower temperature. It also mentions that application of a uniform pressure at lower temperatures is a means of developing more uniform micro-cracks such that a more uniform exfoliation can be obtained when the pressure is released and heat is applied. In this document, the applied pressure is a means of obtaining uniform micro-cracks but does not provide any information about increased weakening of the implanted zone by an increase in the size of micro-cracks. Thus, in this document, exfoliation is achieved by releasing the pressure and applying heat at a temperature a priori greater than the temperature used when the pressure was applied. In this document, unlike the case in this invention, the applied pressure is not used to increase weakening of the implanted zone and therefore to reduce the thermal budget and/or mechanical forces in order to obtain the thin film. Furthermore according to this invention, separation may be achieved under pressure. Moreover, according to one advantageous embodiment of the invention, the applied pressure may be adjusted during the process depending on changes to the gaseous phases present in the micro-cavities.

Gaseous compounds for the purposes of this description means elements, for example hydrogen or rare gases, either in their atomic form (for example H) or in their molecular form (for example H_2) or in their ionic form (for example H^+, H^+_2) or in their isotopic

form (for example deuterium) or in isotopic and ionic form.

Furthermore, ionic implantation refers to any type of introduction of the previously defined compounds alone or in combination, such as ionic bombardment, diffusion, etc.

Regardless of the type of solid material, thermal energy causes coalescence of micro-cavities or micro-cracks which causes weakening of the structure at the micro-cavities layer. This weakening enables separation of the material under the effect of internal stresses and/or pressure in the micro-cavities, and this separation may be natural or may be assisted by the application of external stresses.

Applying pressure is a means of causing coalescence of micro-cavities while preventing the formation of blisters on the plane face. This pressure depends on the state of stress in the implanted zone.

Partial separation means separation in which attachment points are left between the thin film and the rest of the substrate.

The said pressure may be a gas pressure and/or a mechanical pressure applied for example by a piston. It may be applied locally or uniformly over the plane face.

The process may also comprise bonding of a thickener on the said plane face, after implantation of gaseous compounds. The thickener may consist of a wafer that may, for example, be bonded with the said plane face by molecular bonding. The thickener may also be composed of a deposit of one or several

materials. Pressure can then be applied through the thickener. This thickener acts as a stiffener. In this case, the pressure that encourages coalescence of micro-cavities and prevents the formation of blisters takes account of the thickener. The thickener can induce stresses on the structure, encouraging the coalescence of micro-cavities.

Advantageously, while some of the micro-cavities are coalescing, the said pressure is adjusted to remain slightly higher than a pressure called the limiting pressure, below which blisters appear on the said plane face and above which blisters do not appear on the said plane face. This avoids applying unnecessarily high pressures.

The limiting pressure changes in time as the coalescence of micro-cavities varies. Thus, the pressure used according to the invention may be the maximum limiting pressure or it may be a limiting pressure that is applied gradually during the process and that varies as a function of the coalescence of the micro-cavities that in particular depends on the thermal budget (time, temperature). Therefore, the limiting pressure depends on the thermal budget. Thus, for example, for annealing a 300 nm Si film and a 5 µm SiO_2 film at 450°C for a given duration, a pressure of the order of a few bars has to be applied in order to achieve separation, whereas if there is no additional pressure (in other words at atmospheric pressure) annealing at more than 470°C is necessary to achieve separation and obtain a film.

Coalescence may be achieved such that the thin film can be separated from the rest of the substrate simply by pulling them apart.

According to another embodiment, the thin film is separated from the rest of the substrate by applying a heat treatment and/or mechanical forces.

The initial substrate may be a substrate that has already been used to make a thin film according to the process. For example, this substrate that has already been used may be polished to provide a new plane face.

The substrate may comprise one or several homogenous and/or heterogeneous layers on the side of the said plane face. It may be composed of a semi-conducting material, at least on the side of the said plane face. It may comprise all or part of at least one electronic device and/or at least one electro-optical device, on the side of the said plane face.

Due to the applied pressure, the invention can result in thinner self supporting films than are possible with a process without pressure. This pressure prevents the relaxation of micro-cavities in the form of blisters and enables these micro-cavities to interact to cause separation.

The invention can also be used to delay separation of the thin film by the use of an additional step that consists of applying additional pressure on the thin film.

Brief description of the drawings

The invention will be better understood and other advantages and specific features will become clearer

after reading the following description given as a non-limitative example with the attached drawings in which:

- figures 1 to 3 diagrammatically illustrate the different steps in the process for making a thin film according to this invention,
- figure 4 is a diagram showing the variation of the pressure applied to the implanted face of a substrate during a step in the process for making a thin film according to this invention, as a function of time.

Detailed description of embodiments of the invention

The principle used in the invention is based on the use of pressure during one or several heat treatments to weaken the implanted zone while preventing the formation of blisters.

Coalescence may be achieved by combining a heat treatment cycle associated with a pressurization cycle in order to increase the weakening phenomenon without creating blisters on the implanted face. The pressure may be a gas pressure. The weakening phenomenon may be continued until complete separation of the two parts of the substrate. The substrate weakening process at the ion implantation depth continues while coalescence is taking place, and it may go beyond the limits possible with a simple heat treatment. The pressure applied on the implanted face of the substrate makes this result possible by preventing the formation of blisters on the implanted face and also by preventing some blisters from bursting as can occur if there is no applied pressure. The result is that the substrate is much weakened along the layer of micro-cavities.

Figures 1 to 3 show transverse views of a semi-conducting substrate to which the process according to the invention is applied.

The semi-conducting substrate 1 has a plane face 2. A plane face means a face for which the average surface is plane. This includes wafers that have a surface micro-roughness with roughness values varying from a few tens of nanometers to several hundred nanometers. The inventors of this invention have demonstrated that implantation through a surface with a micro-roughness with an RMS (root mean square) value of 10 nm, for example, does not disturb the weakening mechanism and the subsequent fracture. This observation is useful, since this roughness is of the order of magnitude of the roughness of the free face of the film after the transfer. Therefore under these conditions, it is possible to recycle the same substrate several times without the need for surface polishing. In some cases, this face may have a topology that will be eliminated during surface preparation, for example by mechanical-chemical polishing.

Figure 1 illustrates the step in which gaseous compounds are implanted. The plane face 2 is bombarded, for example by hydrogen ions, as described in document FR-A-2 681 472. This ionic bombardment is illustrated by arrows 3. This thus creates a micro-cavities layer 4.

The process according to the invention may also comprise an operation to thicken the required thin film. For example, after the implantation step, it

would be possible to add a wafer on the implanted face of the substrate, by molecular bonding or by another type of bonding processes. This can be done using equipment that puts the substrate and the wafer into contact in a pressurized chamber. Pressure can then be applied on the plane face of the substrate while the thickening wafer is being bonded.

This thickening operation may advantageously be done using a process derived from the process described in document FR-A-2 739 671. For example, a set of materials can be deposited on the plane face of the substrate to make it more rigid. These deposits may be epitaxial or heteroepitaxial growths or deposits of amorphous or polycrystalline materials. For example silicon may be deposited on the plane face of a previously implanted substrate. The added material may be qualified as a thickener, regardless of whether it is bonded or deposited.

For given experimental conditions (materials, ions, dose, energy, implantation and annealing temperature), there is a limiting value for the pressure applied to the plane face of the substrate, P_{limit}, for each thickness of the thin film (thickened or not thickened), below which blisters will appear on the plane face and above which blisters will not appear on the plane face. For example, P_{limit} is equal to 20 bars for a total silicon thickness equal to 2 µm and is equal to atmospheric pressure for 5 µm of silicon. Therefore, when performing the coalescence step according to the invention, it is possible to adjust the pressure while this step is being carried out so

that the pressure remains close to P_{limit}. This avoids the application of unnecessarily high pressures.

P_{limit} also depends on the remaining quantity of gaseous compounds added during the ionic implantation. This quantity of gas may vary with time due to gas diffusion, particularly due to temperature, and due to the fact that micro-cavities that contain this gas become larger. The limiting pressure is a means of avoiding the formation of blisters, but it must not limit the growth of cavities or micro-cracks present close to the implantation depth. When the size of the micro-cracks increases, the same quantity of gas occupies a larger volume and consequently P_{limit} reduces. It is thus possible to determine a step in which coalescence is carried out such that the pressure exerted and the limiting pressure each follow a cycle that begins and ends at atmospheric pressure. The actually applied pressure remains higher than or equal to the limiting pressure. Therefore a weakened layer is recovered at the end of the cycle, at atmospheric pressure.

Under some conditions, the substrate can be fully separated into two parts during the coalescence step. In this case, the cycle is terminated.

Figure 2 illustrates the micro-cavities coalescence step due to the addition of thermal energy T and application of pressure P. For example, the applied pressure corresponds to the cycle in the diagram in figure 4, representing the variation of the pressure P as a function of time t. The applied pressure follows the atmospheric pressure (P_{atm}) –

limiting pressure (P_{limit}) - atmospheric pressure (P_{atm}) cycle. Micro-cavities tend to coalesce to form micro-cracks 5.

Figure 3 illustrates the separation step after which a thin film 6 is detached from the rest of the substrate 1. Two cases can arise after the previous step; firstly the film may be not entirely separated from the substrate, or it may be entirely separated from it.

The process may be carried out such that the thin film is not entirely separated from its original substrate; In this case, the thin film may for example be recovered by means of a stiffening support as described in document FR-A-2 681 472, that is fixed to the implanted face of the substrate. With this invention, this recovery is easier since the implanted zone is more weakened. This means that the thermal budgets necessary are lower and/or the necessary tear off energy is lower. The advantage of a lower thermal budget (time and/or temperature) is the possibility of combining materials with different coefficients of thermal expansion. The advantage of a lower tear off energy is the possibility of choosing a lower bond energy of the stiffener, to facilitate subsequent separation of the thin film and the stiffener according to the description given in document FR-A-2 725 074.

For example, this stiffening substrate may be a silicon wafer, or a flexible film for example made of polymer or ceramic. The wafer may be bonded onto the stiffener by means of a glue or molecular bonding,

possibly by means of an interface layer for example such as an SiO_2 layer.

The process may be carried out such that the thin film is completely separated from its original substrate. Bonding of a stiffening support may not always be necessary. A self supporting film can be obtained as described in document FR-A-2 738 671. However, thin films according to the invention can be obtained with much lower thicknesses. For example, in the case of mono-crystalline silicon, the minimum energy necessary to achieve ionic implantation according to document FR-A-2 738 671 is 500 keV. According to the invention, application of a pressure of 20 bars can lower the minimum implantation energy (to avoid the use of a stiffener) to about 150 keV. Standard implanters can then be used.

As an example, we will now describe how a silicon film is obtained according to this invention. The plane face of a silicon substrate is bombarded by protons at a dose that could cause the appearance of blisters on the bombarded face during a heat treatment at 500°C. This dose may be of the order of 10^{17} cm^{-2} for an implantation energy of 150 keV. In a first phase, a conventional heat treatment is carried out to activate the micro-cavity growth mechanism (for example at 250°C for 2 hours). It is not necessary to apply pressure in this first phase, since coalescence of the micro-cavities is not sufficient to cause the formation of detectable blisters; the limiting pressure is less than or equal to the atmospheric pressure. In a second phase, the implanted face of the substrate is

pressurized (20 bars) and the temperature is increased from 300 to 400°C in 15 minutes and is then kept constant for one hour. The result is total separation between the two parts of the substrate. The temperature is then lowered and the pressure is reduced to atmospheric pressure. The thin film can then be recovered.

If a stiffening support is used, the implanted zone is weakened under pressure and the temperature is reduced to lower the pressure induced by the amount of gas and the temperature. It is necessary to allow for the fact that reducing the temperature causes a significant reduction in the internal pressure in the micro-cavities or the micro-cracks.

This process according to the invention has many advantages. It is a means of obtaining fractures with lower roughnesses than are possible using processes according to known art. This can reduce the thickness that may need to be removed by polishing, for example when making Silicon On Insulator (SOI) substrates. Polishing introduces a dispersion in the thickness that depends on the removed thickness, consequently the invention can be used to make SOI substrates with a more homogeneous thickness. Furthermore, the disturbed zone after the fracture is lower, consequently the invention can reduce the number of residual defects in the thin film.

The thickening possibility makes it possible to make thicker films, for example about 10 micrometers or thicker. These thin films may be used to produce thick SOI structures for making power devices or for making

substrates for the production of "thin layers" of solar cells.

With the process according to the invention, the original substrate can be used several times, possibly after polishing the face of the substrate revealed after the thin film has been detached.

The process is applicable to semi-conducting materials and to other mono-crystalline or other materials.

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CLAIMS

1. Process for making a thin film starting from a substrate of a solid material with a plane face comprising:

- the implantation of gaseous compounds in the substrate to make a layer of micro-cavities at a depth from said plane face corresponding to the thickness of the required thin film, the gaseous compounds being implanted under conditions that could weaken the substrate at the layer of micro-cavities,
- partial or total separation of the thin film from the rest of the substrate, this separation comprising a step in which thermal energy is added and pressure is applied to the said plane face.

2. Process according to claim 1, wherein said pressure is a gas pressure.

3. Process according to claim 1, wherein said pressure is a mechanical pressure.

4. Process according to claim 3, wherein said mechanical pressure is generated using a piston.

5. Process according to claim 1, wherein said pressure is applied locally on the said plane face.

6. Process according to claim 1, wherein said pressure is applied uniformly on the said plane face.

7. Process according to claim 1, wherein it also comprises bonding of a thickener onto said plane face, after implantation of the gaseous compounds.

8. Process according to claim 7, wherein the thickener is composed of a wafer.

9. Process according to claim 8, wherein the wafer is bonded by molecular bonding with the said plane face.

10. Process according to claim 7, wherein the thickener is formed by deposition of one or several materials.

11. Process according to claim 7, wherein said pressure is applied through the thickener.

12. Process according to claim 1, wherein said pressure is adjusted during the coalescence of at least part of the micro-cavities, to remain slightly above a pressure called the limiting pressure, below which blisters appear on said plane face and above which blisters do not appear on said plane face.

13. Process according to claim 1, wherein coalescence is performed such that the thin film is separated from the rest of the substrate by simply pulling them apart.

14. Process according to claim 1, wherein the thin film is separated from the rest of the substrate by application of a heat treatment and/or mechanical forces.

15. Process according to claim 1, wherein the substrate used as the initial substrate is a substrate that has already been used to produce a thin film according to said process.

16. Process according to claim 15, wherein the previously used substrate is polished to provide a new plane face.

17. Process according to claim 1, wherein the substrate supports one or several homogeneous and/or heterogeneous layers on the side of said plane face.

18. Process according to claim 1, wherein the substrate is composed of one semi-conducting material, at least on the side of said plane face.

19. Process according to claim 1, wherein the substrate comprises all or part of at least one electronic device and/or at least one electro-optical device, on the side of said plane face.

20. Process according to claim 1, wherein the separation of the thin film is delayed by the application of an additional step that consists of applying an additional pressure onto the thin film.

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ABSTRACT

PROCESS FOR MAKING A THIN FILM BY APPLYING PRESSURE

The invention relates to a process for making a thin film starting from a substrate (1) of a solid material with a plane face (2) comprising:

- the implantation of gaseous compounds in the substrate (1) to make a layer of micro-cavities (4) at a depth from the said plane face (2) corresponding to the thickness of the required thin film, the gaseous compounds being implanted under conditions that could weaken the substrate at the layer of micro-cavities,
- partial or total separation of the thin film from the rest of the substrate (1), this separation comprising a step in which thermal energy is added and pressure is applied to the said plane face (2).

Figure 2

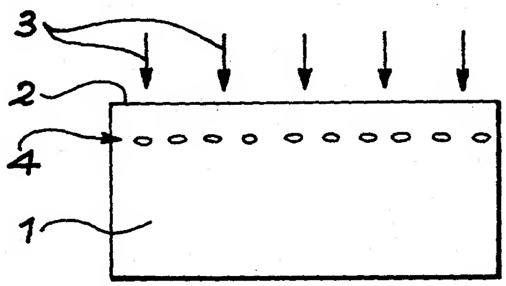

FIG. 1

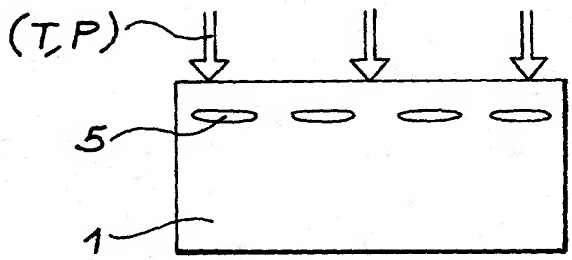

FIG. 2

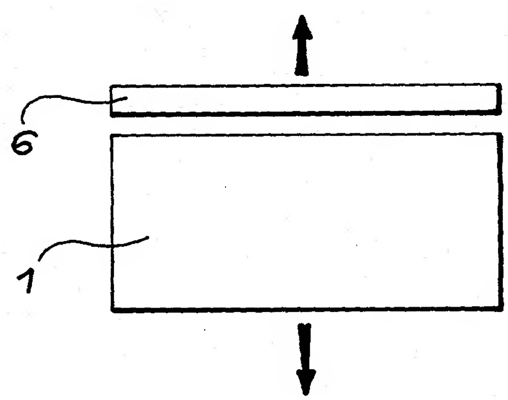

FIG. 3

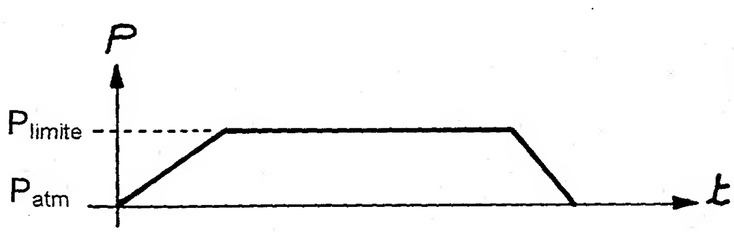

FIG. 4

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Declaration, Power Of Attorney and Petition

WE (I) the undersigned inventor(s), hereby declare(s) that :

My residence, post office address and citizenship are as stated below next to my name,

We (I) believe that we are (I am) the original, first, and joint (sole) inventor(s) of the subject matter which is claimed and for which a patent is sought on the invention entitled

PROCESS FOR MAKING A THIN FILM BY APPLYING PRESSURE

the specification of which

☐ is attached hereto.

☐ was filed on

as Application Serial No.

and amended on

☒ was filed as PCT international application

Number PCT/FR00/01828

on June 29, 2000

and was amended under PCT Article 19

on June 15, 2001

We (I) hereby state that we (I) have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

We (I) acknowledge the duty to disclose information known to be material to the patentability of this application as defined in Section 1.56 of Title 37 Code of Federal Regulations.

We (I) hereby claim foreign priority benefits under 35 U.S.C. § 119 (a)-(d) or § 365 (b) of any foreign application(s) for patent or inventor's certificate, or § 365 (a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed. Prior Foreign Application (s)

Application No.	Country	Day/month/Year	Priority Claimed
99 08379	FRANCE	30 JUNE 1999	☒ YES ☐ NO
			☐ YES ☐ NO
			☐ YES ☐ NO
			☐ YES ☐ NO

We (I) hereby claim the benefit under Title 35, United States Code, § 119 (e) of any United States provisional application(s) listed below.

(Application Number) (Filing Date)

(Application Number) (Filing Date)

We (I) hereby claim the benefit under 35 U.S.C. §120 of any United States application(s), or § 365(c) of any PCT International application designating the United States, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. § 112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR § 1.56 which became available between the filing date of prior application and the national or PCT International filing date of this application.

Application Serial No.	Filing Date	Status (pending, patented, abandoned)

And we (I) hereby appoint : BLAIR Charles H., BRENNAN Michael P., BROCK Christopher M., BUDDE Anna M., BURRIS Kelly K., CANTOR Bernard J., CARLSON Richard L., DESCHERE Linda M., DONLEY Garrett C., ELCHUK Mark D., ERJAVAC Stanley M. , EUSEBI Christopher A., FALCOFF Monte L., FOSS Stephen J., FRENTRUP Mark A., FULLER Roland A., GIBBS Barbara S., GAMBRELL Myriah M., HALLIN Thomas H., HARRIS Gordon H., JORDAN B. Delano, KELLER Paul A., KICZEK Casimir R., LAFATA Joseph L., LALONE Douglas P., MACINTYRE Timothy D., MALINZAK Michael, MASSEY Ryan W., MCCLAUGHRY David A., MILLER H. Keith, MILLER John A., MOUSTAKAS George D., NOLAN Robert S., O'DELL Elizabeth D., OLSON Stephen T., PAPP Joseph R., RETTIG Phillip E., SCHIVLEY G. Gregory, SCHMIDT Michael J., SCHOOF George T., SIMINSKI Robert M., SMIRMAN Preston H., SNYDER Jeffrey L., SOSENKO Eric J., SOTIRIOU Evan A., STEPHENSON James E., STEVENSON Joseph L., STOBBS Gregory A., TAYLOR W.R. Duke, TELSCHER Rudolph A., UTYKANSKI David P., WADE Bryant E., WALKER Donald G., WALLACE Robert J., WALSH Joseph E., WANGEROW Ronald W., WARNER Richard W., WHEELOCK Bryan K., WIGGINS Michael D., ZALOBSKI Michael D., our (my) attorneys, with full powers of substitution and revocation, to prosecute this application and to transact all business in the Patent Office connected therewith; and we (I) hereby request that all correspondence regarding this application be sent to the firm of HARNESS, DICKEY & PIERCE, P.L.C.. whose Address is . P.O. Box 828, Bloomfield Hills, Michigan 48303.

We (I) declare that all statements made herein of our (my) own knowledge are true and that all statements made on information and belief are believed to be true ; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such wilful false statements may jeopardise the validity of the application or any patent issuing thereon.

1-00

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Signature of Inventor

Date

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Post Office Address : The same as residence